

CLAIMS

1. Process for purifying caprolactam, said process comprising
- 5 (a) subjecting the caprolactam to a hydrogenation by treating the caprolactam with hydrogen in the presence of a heterogeneous nickel containing hydrogenation catalyst, and
- (b) distilling at least a portion of the hydrogenated caprolactam in a distillation column, characterized in that the distillation column contains nickel in an
- 10 amount sufficiently low such that  $\Delta\text{PAN}_{\text{Ni}} \leq 3$ , wherein
- $\Delta\text{PAN}_{\text{Ni}} = \Delta\text{PAN} - \Delta\text{PAN}_{\text{Ni}=0}$ ,
- $\Delta\text{PAN}$  = increase of the PAN number of caprolactam during distilling,
- $\Delta\text{PAN}_{\text{Ni}=0}$  = increase of the PAN number of caprolactam during distilling under the same conditions in a distillation column free of nickel.
- 15 2. Process according to claim 1, wherein the distillation column contains nickel in an amount sufficiently low such that  $\Delta\text{PAN} \leq 3$ .
3. Process according to claim 1, wherein the distillation column contains nickel in an amount sufficiently low such that  $\Delta\text{PAN}_{\text{Ni}} \leq 2$ .
4. Process according to claim 3, wherein the distillation column contains nickel in
- 20 an amount sufficiently low such that  $\Delta\text{PAN}_{\text{Ni}} \leq 1$ .
5. Process according to any one of claims 1-4, wherein said distilling is performed continuously.
6. Process according to any one of claims 1-5, wherein the process further comprises, prior to said distilling, separating nickel from hydrogenated
- 25 caprolactam.
7. Process according to claim 6, wherein said separating is effected using filtration.
8. Process according to any one of claims 1-7, wherein the nickel containing hydrogenation catalyst is a fixed bed catalyst.
- 30 9. Process according to any one of claims 1-7, wherein the hydrogenation is a slurry phase hydrogenation wherein nickel containing hydrogenation catalyst particles are suspended in the caprolactam to be hydrogenated.
10. Process according to claim 9, wherein after said hydrogenation the catalyst particles are separated from the hydrogenated caprolactam.
- 35 11. Process according to claim 6 and 10, wherein said separating of nickel from

hydrogenated caprolactam is carried out after said separating of catalyst particles from the hydrogenated caprolactam.

12. Process according to any one of claims 1-11, wherein the amount of nickel in the hydrogenated caprolactam entering said distilling is less than 10 ppm.
- 5 13. Process according to claim 12, wherein the amount of nickel in the hydrogenated caprolactam entering said distilling is less than 1 ppm.
14. Process according to any one of claims 1-13, wherein said distilling is effected in a distillation column having a bottom temperature of between 110 and 180 °C.
- 10 15. Process according to any one of claims 1-14, wherein said distilling is effected in a distillation column in which the caprolactam of the hydrogenated caprolactam has a residence time higher than 5 minutes.
16. Process according to any one of claims 1-15, wherein said distilling is performed continuously and  $\Delta\text{PAN}_{\text{Ni}} \leq 3$  during a period of at least 6 months.
- 15 17. Process according to any one of claims 1-15, wherein the amount of nickel in the hydrogenated caprolactam entering said distilling is sufficiently low such that  $\Delta\text{PAN}_{\text{Ni}} \leq 3$  during a period of at least 6 months.
18. Process according to any one of claims 1-17, wherein water is separated from the hydrogenated caprolactam prior to said distilling.
- 20 19. Process according to claim 1-18, wherein separating nickel from hydrogenated caprolactam is effected after separating of water and prior to said distilling.
20. Process according to any one of claims 1-19, wherein the caprolactam entering said hydrogenation is obtained by rearrangement of cyclohexanone oxime with sulfuric acid or oleum.
- 25 21. Process, for instance according to any one of claims 1 to 20, said process comprising:
- (a) subjecting the caprolactam to a hydrogenation by treating the caprolactam with hydrogen in the presence of a heterogeneous nickel containing hydrogenation catalyst; and
- 30 (b) distilling at least a portion of the hydrogenated caprolactam in a distillation column,
- characterized in that the amount of nickel in the hydrogenated caprolactam entering said distilling is less than 50 ppm, preferably less than 10 ppm, more preferably less than 1 ppm, even more preferably less than 500 ppb and even
- 35 more preferably less than 100 ppb.